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Development of a Water-Base,
Fire-Resistant Hydraulic Fluid

Assignment 81 116
MEL R&D Phase Report 261/66
August 1966

By
C. L. Brown, T. N. Cornish, and
J. A. Marzani

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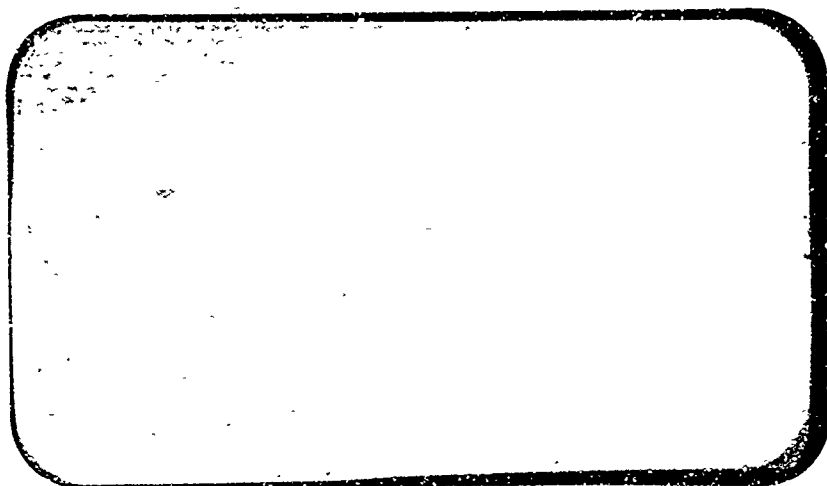
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Transmittal of

1. Transmitted herewith is MEL Research and Development Phase Report 261/66, Development of a Water-Base, Fire-Resistant Hydraulic Fluid. The fluid described herein is LF-4713, a formulation prepared under Contract NObs-90267 by the American Oil Company.

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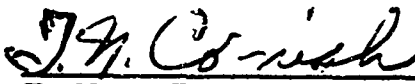
- a. Laboratory studies to determine the cause of thermal instability of Fluid LF-4713.
- b. Improve or replace unstable component of fluid by synthesis or use of additives.
- c. Evaluate performance characteristics of improved form / ation against target objectives.
- d. Samples of new fluids to be supplied upon request to MEL for fire resistant and other performance studies.

Development of a Water-Base,
Fire-Resistant Hydraulic Fluid

Assignment 81 116
MEL R&D Phase Report 261/66
August 1966


By
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ABSTRACT

A corrosion-inhibited, low-pour-point, water-base fluid formulation, LF-4713, containing a soluble, fire-resistant, phosphonated polyethylene thickener, developed under NAVSEC Contract NObs-90267 by the American Oil Company for potential use in hydraulic systems, has been evaluated.

The fluid appears to have good fire resistance and meets many of the target requirements for a satisfactory hydraulic fluid. Its thermal instability and its tendency to form stable foams and to allow corrosion of ferrous metals in vapor spaces require correction.

ADMINISTRATIVE INFORMATION

The study of Fluid LF-4713, a water-base, fire-resistant hydraulic fluid, developed by the American Oil Company under Contract NObs-90267, was conducted for NAVSEC (Code 6634D) under Sub-project S-F020 93 02, (formerly S-R001 03 01, Task 0606), and MEL Assignment 81 116.

This is a report of the current state of development of Fluid LF-4713.

Reports pertaining to this fluid were made by the contractor in references (a) and (b).

REFERENCES

- (a) Bunting, K. R., et al, "Development of Nonflammable Hydraulic Fluid," final rept, Contract NObs-90267, 1 Apr - 1 Jun 1965
- (b) Wazeter, Francis X., "Acute Toxicity Tests of LF-4713, Development of Non-flammable Hydraulic Fluid," Contract NObs-90267, 8 Jul 1965

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DEVELOPMENT OF A WATER-BASE, FIRE-RESISTANT HYDRAULIC FLUID

1.0 INTRODUCTION

This report describes the evaluation of a new water-base fluid to determine its potential for further development into a water-base, fire-resistant hydraulic fluid. This work was accomplished as part of a program aimed at obtaining a hydraulic fluid containing water and a fire-resistant, nonaqueous thickener. The synthesis and formulation of the fluid were done by the American Oil Company under NAVSEC (formerly BUSHIPS) Contract NObs-90267. Preliminary fluid performance studies were done by the American Oil Company and by experiments conducted at this Laboratory.

2.0 FLUID FORMULATION

The fluid under study, LF-4713, is that formulation developed under NAVSEC Contract NObs-90267 that most nearly meets the target objectives. It has the composition shown in Table 1.

Table 1
Composition of Fluid LF-4713

Component	Function	Concentration, %
Potassium hydroxide neutralized, phosphonated polyethylene ¹	Thickener	27
Potassium bis-hydroxymethyl phosphonate	Pour-point depressant	30
Potassium molybdate	Corrosion inhibitor	2
Water	Base fluid	41

¹Made from 12,000 molecular weight polyethylene.

A 2-quart sample submitted to this Laboratory for study was identified as Sample "LF-4713."

3.0 TARGET OBJECTIVES

Contract NObs-90267 establishes the following objectives:

A. A fire-resistant water base fluid for shipboard hydraulic system use is required. This can be either a solution, an emulsion or a suspension. The non-aqueous residue of such fluid shall also be fire-resistant.

B. Desired Properties.

1. Fire Resistance. Flash points above 450 F., * fire points above 550 F. and autogenous ignition temperature above 900 F. are optimum. It should be emphasized that, all other conditions being equal, the fluid showing greatest fire resistance in the non-aqueous portion of the fluid would be considered the more desirable.

2. Viscosity shall be 850 c. s. maximum at 25 F and between 25 - 31 c. s. at 150 F.

3. Pour Point - The pour point shall be 0 F maximum

4. Shear Stability - The viscosity after shearing shall not show more than $\pm 10\%$ change at 150 F.

5. Specific Gravity - The specific gravity at 60/60 F shall not exceed 1.6. It is to be noted that specific gravities of the order of 1.0 to 1.2 are optimum.

6. Lubricating Ability - The fluid shall be capable of lubricating shipboard hydraulic pumps to pressures of 5000 psi. Pumps now in use include screw types and variable stroke piston types.

7. Compatibility -

a. Metals - The fluid shall be compatible with the alloys in the submarine hydraulic systems. The following are the metals.

Steel	Bronze
Copper	Phos. Bronze
Copper Nickel	Aluminum
Nickel Copper	(All Aluminum in use is anodized)

* Abbreviations used in this text are from the GPO Style Manual, 1959, unless otherwise noted.

b. Elastomer - The fluid shall be compatible with Buna N rubber.

c. Sea Water - The fluid shall be functional with 10% sea water contamination.

8. Stability - The fluid shall be stable under conditions of storage (-20 to +110 F) and use (+25 to 180 F), and filterable without degradation of properties through 5 micron rated porosity filters.

9. Foaming - The fluid shall not produce stable foams.

10. Toxicity - The fluid shall be formulated to minimize hazards resulting from its use.

11. Fluid Residues - Fluid residues shall be removable by flushing with water.

4.0 APPROACH AND METHODS

Experiments conducted in this Laboratory were aimed at supplementing experiments conducted by the American Oil Company, reference (a). The basic objective was to determine the key performance characteristics of Fluid LF-4713 to assess the merits and deficiencies of the present fluid formulation and to determine the merits of additional formulation work. However, Laboratory experiments were restricted by the sample size. The experiments conducted at this Laboratory are described below.

4.1 Fire Resistance. High-pressure combustion measurements in a closed system in the absence of a flame were obtained in a static combustor under development in this Laboratory. A description of the apparatus and procedure used are given in Appendix A. Fluid combustion characteristics in an open system at atmospheric pressure upon exposure to an open flame (crucible test) were determined by applying a Meeker-type gas burner flame to the fluid in a porcelain crucible and observing the relative ease of ignition of vapors by the flame and the tendency of the fluid's heat of combustion to sustain combustion. The autoignition temperature by ASTM Method D2155-63T also was determined.

4.2 Compatibility. Compatibility of the fluid with seawater was determined by observing the effect of adding 10-percent ASTM D-665 synthetic seawater, the formation of insoluble reaction products in the fluid after storage at 150 F for 48 hours indicating incompatibility. Compatibility with paint was determined by observing a painted surface for evidence of softening, blistering, or peeling when covered with a 0.05-ml drop of test fluid for 10 days at ambient temperature and at 140 F. The paint surface was prepared by applying a 2-mil wet coat of red lead primer (MIL-P-17545B) on a 4- x 12-inch steel panel and curing it for 24 hours at 140 F. A 2-mil wet coat of grey alkyd-type inside deck paint (JAN-P-700) was then applied over the primer and cured for 24 hours at 140 F.

4.3 Removability of Dried Residue. Removability of dried fluid residue was determined on a sample contained in a porcelain dish. Drying of the fluid was achieved by boiling vigorously to evaporate the water component. Ease of removing residue was determined by wiping the dish surface with a water-moistened rag.

4.4 Stability in Presence of Copper at Elevated Temperatures. The stability of the fluid in presence of copper was determined by subjecting the fluid to the MIL-H-19457B (SHIPS) hydrolytic stability test. The test consists of heating the fluid to 200 F in presence of a copper strip for 48 hours. Since the fluid as formulated contains water, no water was added as is done in the test as written in the specification.

4.5 Other Properties. All other properties studied were determined by methods shown in Table 2.

Table 2
Test Methods

Fluid Property	Method
Viscosity	ASTM D-445
Foaming tendency	ASTM D-892
Neutralization number	Method 5105.3 ⁽¹⁾
Shear stability (sonic shear)	(2)
Compatibility with elastomeric materials	Method 3603.3 ⁽¹⁾
Static corrosion	MIL-H-19457B
Rust inhibition	ASTM D-665
Mean Hertz load	Method 6503 ⁽¹⁾
Four-ball wear	Method 6503 ⁽¹⁾ as modified by MIL-L-17331D and run at 75-80F

¹Method found in Federal Test Method Standard 791a.

²Proposed Method of Test for Shear Stability of Polymer Containing Oils, "ASTM Standards on Petroleum Products and Lubricants, 1961, Appendix XII, Page 1160.

5.0 RESULTS OF STUDIES

All Marine Engineering Laboratory results appear in Appendix B. Some of the performance characteristics determined by the American Oil Company and intended to supplement those determined by this Laboratory also appear in Appendix B. Performance characteristics of three current service hydraulic fluids, (MIL-L-17331D (SHIPS), MS 2190-TEP petroleum oil; MIL-H-19457B, Type 1 triaryl phosphate fire-resistant fluid; and MIL-H-22072 (AER) water-glycol-type fire-resistant fluid) are shown for comparison with Fluid LF-4713. Target objectives for the fluid are also shown in Appendix B.

6.0 DISCUSSION OF FLUID PROPERTIES

The data developed indicates that Fluid LF-4713 meets a number of the objectives for a fire-resistant hydraulic fluid. However, certain of the fluid's performance characteristics require improvement, while others need to be studied in more depth before a decision as to the significance of the property can be reached. Specific fluid performance characteristics are discussed below.

6.1 Fire Resistance. The high-pressure fire resistance of Fluid LF-4713 in the static combustor, as measured by its minimum self-ignition temperature (MSIT) of 775 F, is superior to that of the petroleum base hydraulic fluid (453 F) and to the two fire-resistant hydraulic fluids, the triaryl phosphate (618 F), and the water-glycol-type fluid (486 F). During the course of the investigation, the pressure rise associated with ignitions of Fluid LF-4713 (at temperature up to 825 F) has been well below that encountered for any of the other hydraulic fluids mentioned above. This low energy release and its high MSIT make Fluid LF-4713 appear good from an explosion hazard standpoint.

6.1.1 While the MSIT of Fluid LF-4713 is 775F, exothermic reactions are observed at temperatures down to 386 F at 2400 psig. (No pressure rises are detected in the 386 to 775 F range.) This value of 386 F, called the minimum reaction temperature (MRT), is comparable to that for the petroleum oil (385 F) and below that of the water-glycol (485 F) and of the triaryl phosphate (500 F). The residue from the reaction of Fluid LF-4713 in the static combustor at the MRT of 386 F, and at 401, 426, and 457 F, was in each case a light-brown, water-soluble solid. The residue from the reaction at 470 F and at higher temperatures was black, relatively water insoluble, and a flaky solid. The relatively low MRT of Fluid LF-4713 is not considered too serious an indication that this is an explosive-type fluid, since the exothermic reactants would have to reach quite a high temperature (775 F) before explosive conditions are achieved.

6.1.2 While it is recognized that spontaneous ignition of this fluid did not occur at temperature below 775 F, indicating a high degree of fire resistance, the observation of exothermic reactions at temperatures as low as 386 F raises the question of the thermal instability of the components at 2400 psi. It could be reasoned that a 1/2-ml sample in the static reactor quickly loses all of its water, that the solid residue is deposited on the hot walls of the reactor, and that the breakdown observed

would have little significance on the properties of a large quantity of the fluid exposed to such a temperature under conditions where water losses would be small. This is partly substantiated by the fact that the residue is readily soluble in water.

In order to provide additional information on the nature of the fluid instability, 80-ml portions of Fluid LF-4713 were subjected to elevated temperatures at an initial pressure of 2400 psi. The fluid was contained in a 100-ml unstoppered conical centrifuge tube. Thermocouples were inserted into the liquid and the vapor space above it. The sample and tube were heated to the test temperature in a steel high-pressure bomb (inside diameter 2 inches, length 10 inches). The bomb was pressurized to 2400 psi at room temperature, and the resulting increase in pressure due to heating was monitored by means of a pressure-sensitive transducer. When the fluid reached the desired test temperature, the bomb was removed from the heat and allowed to cool to room temperature (usually overnight), and the fluid sample was removed to measure its viscosity and neutralization number. The viscosity and neutralization number were compared to an untreated sample of the fluid. The data collected in this manner are tabulated in Table 3.

Table 3

High-Pressure Stability Studies

Liquid Phase		Vapor Phase		Values after Exposure		
Maximum Temperature F	Time min	Maximum Temperature F	Time min	Maximum Pressure psig	Viscosity 100 F	Neutralization Number
Room	-	Room	-	-	64.3	17
300	60	300	56	3600	38.6	15
345	74	335	68	3800	39.0	21
440	110	-	-	4250	37.3	22

Samples heated at the three test temperature showed little or no color change, a very small water loss, and less than 0.5-percent solids. Considerable vapor space corrosion was noted at the 440 F test temperature. The time listed in Table 3 is the time required to reach the test temperature. The pressure is the maximum pressure observed at the test temperature.

It is admitted that the test conditions were rather severe in terms of time duration of exposure. These data, nevertheless, indicate a marked reduction in the fluid viscosity upon exposure to temperatures as low as 300 F. The neutralization number was not significantly affected at this temperature. This latter fact suggests that the loss in viscosity is due primarily to a thermal polymeric rupture rather than an oxidative breakdown. The instability and its effect on performance characteristics require further study.

6.2 Viscosity. Fluid LF-4713 met the 25 and 150 F viscosity objectives for unused fluid. Viscosity changes observed during performance studies are discussed elsewhere in this report.

6.3 Specific Gravity. The specific gravity of Fluid LF-4713 (1.4 grams/ml) is below the 1.6 maximum limit imposed by current hydraulic system configuration, but is beyond the optimum range of 1.0 to 1.2.

6.4 Foaming Tendency. The stability of the foam generated in the foaming tendency tests and during the Mean Hertz Load determinations indicates the need for lowering the surface energy of Fluid LF-4713. Such a lowering was provided by the inclusion of an antifoam agent into an earlier formulation, reference (a).

6.5 Pour Point. Fluid LF-4713 met the pour-point objective. As indicated by cloud point, there is no evidence of crystallization before the pour point is reached.

6.6 Shear Stability. The fluid may not be completely stable to shearing. A discrepancy exists between the viscosity changes of the fluid after sonic irradiation as measured by this Laboratory immediately after shearing in a closed system (8.3% increase) and the viscosity changes obtained by the American Oil Company (1.6% decrease). Both laboratories subjected the fluid to approximately the same degree of sonic energy.

6.7 Compatibility. Fluid LF-4713 was found to be compatible with 10-percent seawater. While a slight cloud formed upon adding seawater to the fluid, it readily redissolved upon stirring. The fluid was found to be compatible with an alkyd-type paint in that it caused no softening, peeling, or blistering after contact with the paint at 75 and 140 F, when the fluid residue was removed from the paint surface while both were still warm. However, when the fluid and painted surface were cooled from 140 F to room temperature, the fluid residue had hardened; and when a spatula was applied to the edge of the residue, the bond between the fluid residue and alkyd paint layer was so strong that the outer coat of paint was removed with the fluid residue, leaving the paint undercoat exposed. The paint compatibility experiment at 140 F was repeated and the residue again cooled to room temperature. When the residue was then wiped with a water-moistened rag, the outer paint coat remained intact and unchanged. With respect to elastomers, the volumetric changes of various materials immersed in the fluid for 7 days at 158 F were very small but always in the direction of a reduction in elastomer volume.

6.8 Removability of Dried Residue. The cooled fluid residue contained in the porcelain crucible was readily removed from the crucible with a water-moistened rag.

6.9 Adhesive Characteristics. Evaporation of water from Fluid LF-4713 at room temperature results in a rather hard, tacky residue. It was observed during the course of the high-pressure, fire-resistance testing that the ground-glass plunger of the hypodermic syringe used to measure the sample had a tendency to freeze when left uncleaned in one position for a period of time. The degree to which the hypodermic plunger adhered to the barrel depended upon the time interval of exposure (at room temperature). It would appear that a system component wetted with a film of the fluid, such as piston pump, solenoid valve, or piston-type accumulator,

could possibly freeze up in a similar manner. Accumulator piston O-rings, which operate in an area lubricated by a film of hydraulic fluid but are vented to the atmosphere, could be a potential trouble spot. This phenomenon will require further exploration.

6.10 Corrosion. No evidence of corrosion was detected in the static corrosion test conducted at this Laboratory. However, corrosion coupons immersed in the fluid during Vickers vane pump performance tests by the American Oil Company indicated that the fluid can cause corrosion of zinc-containing components, such as galvanized steel, reference (a). In a dynamic test at 200 F with a copper strip (MIL-H-19457 hydrolytic-stability test without addition of water), the attack on copper was no greater than allowable with triaryl phosphates, and the neutralization number change of 1.1, recorded in this test, is within the repeatability limit of the neutralization number method used.

6.11 Rust Inhibition. Fluid LF-4713 was found to be sufficiently rust inhibited to prevent rusting of a polished 1018 steel specimen immersed in the fluid, when the fluid was contaminated with 10-percent synthetic seawater and stirred for 48 hours at 140 F. After this experiment half of the fluid and the metal specimen were removed, and a longer steel specimen was set in the test cell so that about half of its length was immersed in the remaining fluid and half exposed to the fluid vapors. Severe rusting of the upper, vapor space, portion of the specimen occurred within 24 hours.

6.12 Lubricating Ability. Fluid LF-4713 has good load-carrying capacity, as indicated by the Mean Hertz load test, since its Mean Hertz load (123 kg) was four times higher than that of the three service hydraulic fluids. Its four-ball wear test scar diameter (0.55 mm) indicates that its wear characteristics, as defined by this procedure, are not as good as that of the petroleum oil (0.27 mm), about the same as that of the triaryl phosphate (0.47 mm), and better than that of the water-glycol-type fluid (0.72 mm). While the results of these bench tests cannot be directly extrapolated to predict fluid performance in service-type machinery, they did not reveal any apparent deficiencies in the lubricating ability of the fluid. In reference (a) the contractor noted that while the fluid had lubricating ability it might be necessary to improve it. The Vickers vane pump test data developed, reference (a), shows that the fluid's lubricating ability was being improved considerably during the contract work. However, comparative pump-wear data were not available to evaluate the significance of the performance of Fluid LF-4713 with respect to service-type hydraulic fluids.

6.13 Storage and Use Stability. No evidence of fluid instability (such as phase separation or additive precipitation) was observed by either the American Oil Company or this Laboratory during heating or chilling in laboratory experiments or during performance runs. The American Oil Company observed no evidence of deposits or filter clogging caused by filtration of the fluid through 5-micron porosity membrane paper and through the 10-micron porosity filters in their Vickers vane pump assembly.

7.0 FLUID DEVELOPMENT POTENTIAL

The concept of a fluid formulation such as Fluid LF-4713 is a promising approach for development of a fire-resistant hydraulic fluid. Fluid LF-4713 meets the fire-resistance requirement and several other target objectives. However, several areas of fluid performance require further exploration and definition.

7.1 Further Work. The areas of fluid performance requiring improvement and further study are shown below.

7.1.1 Thermal stability at pressures up to 3000 psi should be measured.

7.1.2 Tendency to form stable foams should be corrected.

7.1.3 Vapor space corrosion inhibition should be provided.

7.1.4 The tendency of the fluid to corrode metals, such as the attack on galvanized steel described by the American Oil Company should be confirmed, and corrected if necessary.

7.1.5 The possible tendency of the fluid to thicken after sonic irradiation requires exploration to establish its limits and significance.

7.1.6 While material compatibility studies did not show excessive swelling or shrinkage in volume, the slight volume decreases detected may indicate leaching of the plasticizers. Thus, materials of interest, such as Buna N, should be subjected to tensile strength, hardness, and elongation measurements after exposure to the fluid; and to dynamic O-ring studies in this Laboratory's hydraulic system test facility.

7.1.7 Bulk modulus of Fluid LF-4713 has not been measured. While water, a major component, has a high bulk modulus, the contribution of the other fluid components to bulk modulus, particularly the phosphonated polyethylene, is unknown. Therefore, the bulk modulus of the fluid at high pressures should be measured.

7.1.8 The tendency of the fluid to freeze components when sliding components wetted by the fluid are exposed to permit evaporation should be explored.

7.1.9 Toxicity of the final fluid formulation should be studied. In this connection the toxicity studies reported by the contractor in reference (b) do not indicate a potentially severe toxicity problem.

7.2 Standardization of Manufacture. In addition to further characterization and improvement of fluid properties, the synthesis and formulation procedures used to prepare it require further study. The ability to prepare replicate batches of fluids with satisfactory quality control in laboratory, pilot plant, and full-scale production quantities should be explored. The ultimate cost of a satisfactory final fluid of this type in service quantities also should be analyzed and a realistic estimate obtained.

8.0 CONCLUSIONS AND FUTURE ACTIONS

It is concluded that the further development of Fluid LF-4713 should be pursued by the following course of action.

8.1 Determine the fluid component or components subject to instability at pressures up to 3000 psi and at elevated temperatures and reformulate to correct this fluid deficiency.

8.2 Evaluate performance characteristics of new formulation to determine performance deficiencies of new formulation requiring correction.

8.3 When laboratory performance studies indicate that the fluid target objectives have been achieved by reformulation, a 150-gallon quantity of the improved fluid should be prepared for further performance-property characterization studies, including experiments in the Laboratory's hydraulic system fluid study facility.

8.4 Quality control and cost studies of laboratory, pilot plant, and full-scale production quantities of the improved, final fluid should be conducted.

Appendix A
High-Pressure Combustion Test Procedure

An apparatus has been developed at the United States Navy Marine Engineering Laboratory to investigate the combustion phenomena of hydraulic-type fluids at elevated pressures. The method of investigation involves a series of runs in a hot pressurized bomb at varying conditions of temperature, pressure, and concentration. The several reaction parameters may be studied in detail by varying them one at a time, while maintaining the others constant. In this report, however, only relative fire-resistance levels at the same conditions of pressure and reactant sample volume have been established for various fluids. It should be emphasized here that variation of the above parameters, as well as surface effects and system geometry in a detailed investigation, will not affect all fluids either in the same direction or magnitude of change of the fire-resistance level.

The apparatus consists primarily of a reaction chamber and a driver gas chamber, Figure 1-A. A pressure differential is established across a quick-opening valve which separates the chambers. This pressure differential is used to inject a measured fluid sample from a sample well in the drive gas chamber in the hot pressurized reaction chamber. The desired temperature and pressure conditions are maintained in the reaction chamber prior to injection. This chamber is a 12-inch cylindrical 304 SS tube of 5/16-inch inside diameter. Three chromel-alumel thermocouples and one strain gage pressure transducer are used to determine steady-state run conditions and to monitor the reaction.

Data is compared on the basis of initial reaction temperature versus delay. There appear to be two temperature levels which are of most concern, represented by the minimum reaction temperature (MRT) and the minimum self-ignition temperature (MSIT). The minimum reaction temperature is defined as that initial temperature below which no temperature or pressure increase above the initially set values is apparent after injection of the test fluid sample. The minimum self-ignition temperature is defined as that initial temperature below which no positive or "hot" ignitions occur. The MSIT positive ignitions are indicated by a abrupt increase in the reaction rate shown by a sharp rise in temperature and/or pressure. Between the MRT and MSIT values there may exist an area where preignition reaction occur but do not propagate into positive ignitions. However, changes such as an increase in sample or a decrease in the heat losses of the system may be sufficient to allow temperatures below the MSIT to promote positive ignitions. The energy required to obtain positive ignition may come either from the initial chamber conditions or exothermic preignition reactions which occur above the MRT. It therefore appears that the MRT should provide a more reliable value for the application of a level of oxidative and/or thermal instability at set conditions which could, under other conditions, be accelerated to an explosion. The explosion hazard may be better associated with the MSIT for set conditions. The relative reactivity of the fluids is indicated by the maximum pressure rise that was observed during the investigation of each fluid, at and above its MSIT. This pressure rise cannot safely be extended beyond the temperature range which was investigated. The ignition delay is taken as the time elapsed from injection to the maximum indicated temperature or pressure, whichever is the shorter.

No corrections of delay time have been applied to compensate for the effect of heat losses or the amount of reaction sustained after maximum values have been attained. These effects are prominent at long ignition delays. Correlation of delay

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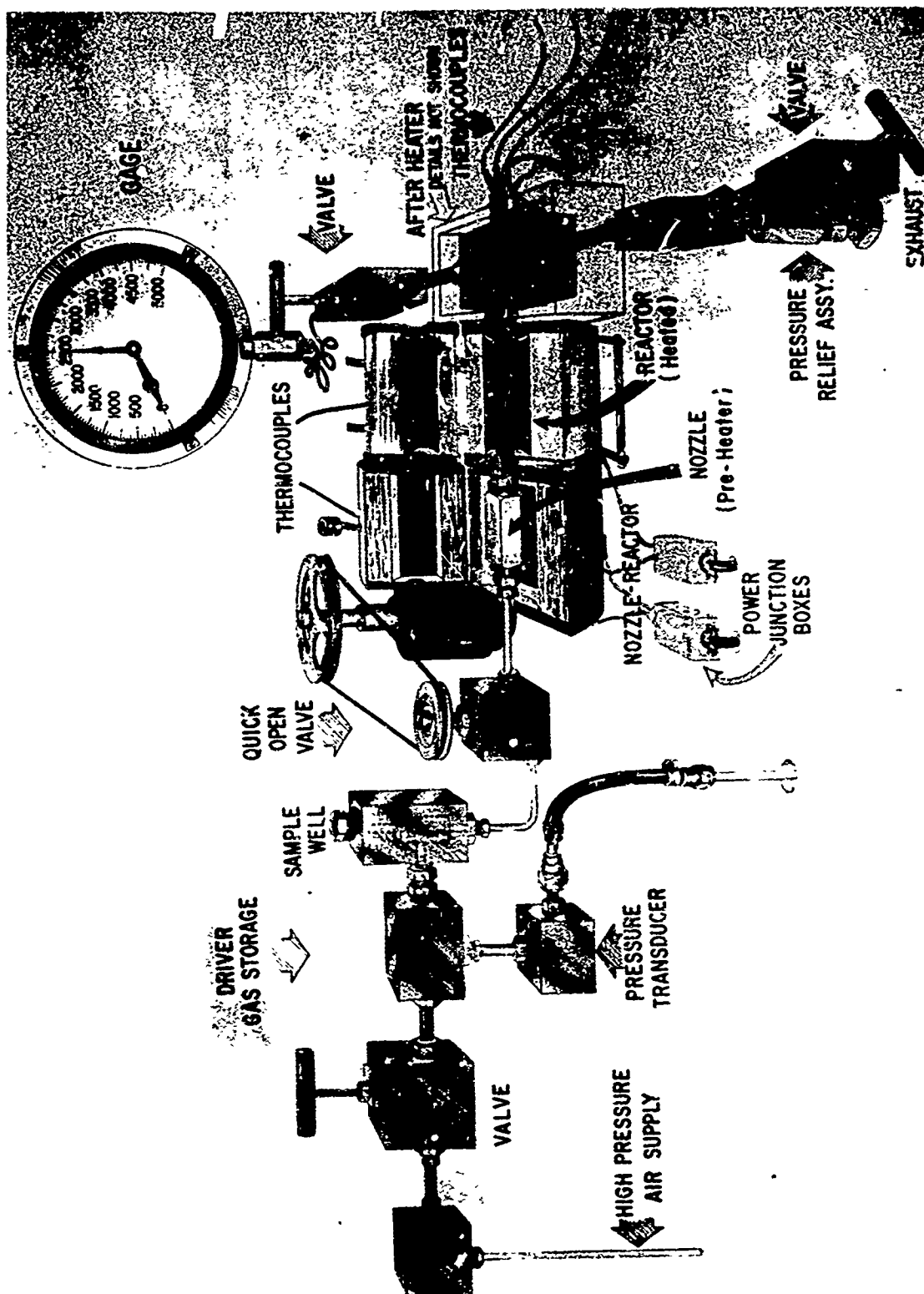


Figure 1-A
Combustion Apparatus

data over a wide combustion range may be further complicated by the occurrence of different reaction mechanisms at the lower temperature associated with long delays.

Test fluid volume used for the fluids shown in this report was 0.5 ml. The initial reactor tube pressure was 2000 psig, and the driving pressure was 3000 psig. The resultant pressure upon injection of sample into the reactor tube was approximately 2400 psig.

Appendix B

Test Results

	Fluid LF-4713 Determined by		Naval Hydraulic Fluids			Contract No. 68-90267 Objective
	Marine Engineering Laboratory	American Oil Company	MIL-L-17331D, MS-2100 TEP Petroleum Oil	MIL-H-19457B, Type I, Triaryl Phosphate	MIL-H-22072 Water-Glycol Type	
Fire resistance						
High-pressure static combustor (2400 psig)						
MSIT minimum self-ignition temperature, F	775	-	453	618	496	-
MRT minimum reaction temperature, F	386	-	385	509	463	-
No reaction temperature, F	370	-	375	490	460	-
Maximum pressure rise observed, psi	55	-	600	350	475	-
Temperature range observed, F	370-775	-	375-506	490-660	470-675	-
Temperature at which maximum pressure rise observed, F	775	-	462	630	675	-
Crucible test	Difficult to ignite ⁽¹⁾	-	Readily Flammable	Difficult to ignite ⁽¹⁾	Residue is readily Flammable	Nonflammable Residue
Autoignition temperature, F	1030	> 1250 ⁽²⁾	745	1040	775	900 min
Flash point, F	-	> 640 ⁽³⁾	415	515	-	450 min
Fire point, F	-	> 640 ⁽³⁾	475	690	-	550 min
Viscosity, centistokes, at F						
25	410 ⁽⁴⁾	659	2000 ⁽⁴⁾	1600 ⁽⁴⁾	265 ⁽⁴⁾	550 max
100	66.8	-	57.5	48.4	44.7	-
130	39.6	-	40.1	21.5	23.5	-
150	30 ⁽⁴⁾	30.2	26 ⁽⁴⁾	14 ⁽⁴⁾	19 ⁽⁴⁾	25-31
ASTM slope, 100-130 F	0.53	-	0.73	0.8°	0.60	-
Specific gravity, g/ml	-	1.4	0.85	1.15	1.05	1.6 max 1.0-1.2 optimum
Foaming tendency, 75 F	-	-	-	-	-	-
Foam after 5-min aeration, ml	420	-	190	< 10	30	No stable foam
Foam after 10-min settling, ml	250	-	0	0	0	-
Foaming tendency, 140 F	-	-	-	-	-	-
Foam after 5-min aeration, ml	600	-	120	< 10	380	No stable foam
Foam after 10-min settling, ml	360	-	0	0	0	-
Pour point, F	-	-50	-10	-5	-40	0 max
Cloud point, F	-	-50	-	-	-	-
pH	-	8	-	-	9	-
Neutralization number	12.4	-	0.16	0.06	-	-
Electrical stability, sonic irradiation (10 kc, C=2 ampere 35 min)	-	-	-	-	-	-
Viscosity change, centistokes at 100 F,	+8.3	-1.6 ⁽²⁾	-0.6	-0.2	+0.5	±10 at 150 F
Compatibility with 10% seawater (48 hr at 150 F)	Satisfactory ⁽⁸⁾	-	Satisfactory	Satisfactory	Precipitate Forms	Must be compatible
Compatibility with elastomeric materials, volume increase,	-	-	-	-	-	-
Buna N	-1.1	-	-0.6	129.2	4.0	Must be compatible
Butyl	-1.2	-	132.6	0.3	0.8	-
Silicone	-0.3	-	4.4	0.3	-	-
Polyvinyl chloride	-0.3	-	-0.2	Formed a gelatinous mass	-	-
Viton B	-0.2	-	-0.1	1.6	14.6	-
Neoprene	-1.4	-	7.4	86.6	-	-
Compatibility with alkyl paint	Satisfactory	-	Satisfactory	Unsatisfactory	Unsatisfactory	Must be compatible
Removability of dried residue	Satisfactory	-	-	-	Satisfactory	Flushable with water

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	Fluid LF-4713 Determined by		Naval Hydraulic Fluids			Contract NObs-90267 Objective
	Marine Engineering Laboratory	American Oil Company	MIL-L-17331D, MS-2190 TEP Petroleum Oil	MIL-H-19457B, Type 1, Triaryl Phosphate	MIL-H-2207C Water-Glycol Type	
Static corrosivity, weight change, mg/cm ²	-	-	-	-	-	-
Aluminum	+0.02	-	-	0	-0.05	Must be compatible with System Metals
Brass	-0.04	-	-	+0.01	+0.01	
Steel	-0.06	-	-	+0.01	0	
Zinc	-0.01	-	-	+0.02	-4.3	
Copper-nickel (90-10)	-0.05	-	-	-0.01	-	
Stability in the presence of copper at 200 F	(5)	-	-	-	(5)	-
Copper specimen weight loss, mg/cm ²	0.24	-	0.02	0.21	0.07	-
Copper specimen appearance	Tarnished	-	Olive drab	Tarnished	Tarnished	-
Neutralization number increase	1.1 ⁽⁶⁾	-	0.01	0.02	-	-
Insolubles	Nil	-	Nil	Nil	Nil	-
Rusting tendency, 48 hr with 10% seawater	-	-	-	-	-	-
Specimen totally immersed in fluid	No rust	-	No rust	No rust	No rust	Functional in presence of sea water
Specimen partially immersed in fluid	-	-	-	-	-	-
Vapor space	Severe rust	-	-	-	-	-
Liquid phase	No rust	-	-	-	-	-
Mean Hertz load, kg	123 ⁽⁹⁾	-	28.2	24.7	32.9	Must lubricate hydraulic pumps
Four-ball wear test (75-80 F scar diameter, mm)	0.55	-	0.27	0.47	0.72	-
Storage and use stability	Satisfactory	Satisfactory	Satisfactory	Satisfactory	-	Satisfactory

¹ Vapors difficult to ignite. Heat input required to maintain continuous flame. LF-4713 was most difficult to ignite.

² Sample prepared by driving off water until bubbling ceased (reference (a) indicates 1450 F in some places; however, Company indicates this in error)

³ No flash or fire observed upon heating for several hours at 220-230 F to drive off water, or when solid residue was heated to 640 F

⁴ Obtained by extrapolation of 100 and 130 F values

⁵ No water added to test cell.

⁶ This increase is within the repeatability of the method

⁷ 10 kc, 0.7 ampere, and 30 min

⁸ White flocculent precipitate formed upon adding seawater. Fluid cleared when stirred. No further precipitate formed when stored.

⁹ Large amount of stable foam observed during test.

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13. ABSTRACT <p>A corrosion-inhibited, low-pour-point, water-base fluid formulation, LF-4713, containing a soluble, fire-resistant, phosphonated polyethylene thickener, developed under NAVSEC Contract NObs-90267 by the American Oil Company for potential use in hydraulic systems, has been evaluated. The fluid appears to have good fire resistance and meets many of the target requirements for a satisfactory hydraulic fluid. Its thermal instability and its tendency to form stable foams and to allow corrosion of ferrous metals in vapor spaces require correction.</p> <p>(author)</p>		

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